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Characterization of Ionomer Compatibilized Blend Morphology Using Synchrotron Small-Angle X-ray Scattering

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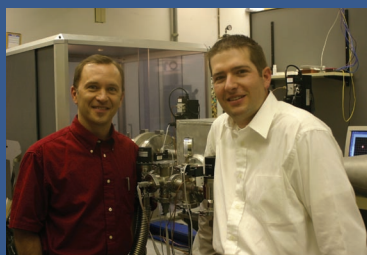
Synchrotron small-angle x-ray scattering (SAXS) was used to characterize the morphology of a non-crystalline polyester/polyamide blend. The blend's components were made compatible via the addition of a sulfonated, polyester copolymer (that is also an ionomer). We used a two-component correlation function to provide detailed information on the size and structure of the phase-separated domains. With increasing ionic functionality, achieved by varying either the ion content or the ionomer loading, the sizes of the dispersed domains in the blends decreased. In addition, SAXS analysis of the 50/50 blend series indicated that the ionic incorporation led to a shift in the morphology to a more elongated, co-continuous structure.

While small-angle laser light scattering (SALS) has often been used to investigate the micro-scale phase separation of polymer blends, small-angle neutron scattering (SANS) and small-angle x-ray scattering (SAXS) are particularly useful in the study of these complex heterogeneous systems due to their ability to probe much smaller size scales. Although SANS has been used to probe information such as the radius of gyration, correlation lengths, and Flory-Huggins interaction parameters of blend components, recent reports have outlined the advantages of using synchrotron SAXS to measure these important blend parameters. The advantages of using SAXS over SANS include: the elimination of isotopic labeling, the high intensity/resolution that allows for fast data collection, and the ability to dynamically examine the phase-separation process in real time.

In the area of blend compatibilization, research efforts using SAXS have been primarily limited to studying the effect of block copolymers on blend morphology and interfacial properties. Here, we describe our efforts to apply synchrotron SAXS to an amorphous polyester (PETG)/polyamide (T40) blend system. This system is compatibilized through the incorporation of an ionomer (known as SPETG) that bears charged groups capable of forming strong, specific intermolecular interactions with the uncharged, polar blend components. In these blends, the specific interactions (**Figure 1**) act at the interface to lower the interfacial tension and thereby lower the domain size of the dispersed phase.

With SAXS, we investigated the morphological features of the blends using the Debye-Bueche scattering theory, employing a two-parameter correlation function. The resulting scattering curves of the binary blends are shown in **Figure 2**. The solid lines represent the nonlinear regression fits (using the inset equation) to the scattering data and show the excellent agreement between the theoretical and experimental curves. As shown in the inset graph, the two-parameter correlation function divides the scattering into contributions from short-range (a_1) and long-range (a_2) fluctuations present in the system. The relative contribution of each is described by the fractional contribution term, f .

In comparison to the blend with pure PETG, the scattering curves for the



Authors (from left) Robert Moore and Greg Gemeinhardt

SPETG/T40 samples show a prominent increase in intensity at small values of q . This scattering behavior indicates a decrease in the dimensions of the dispersed phase with the incorporation of functional groups capable of forming intermolecular interactions. From the fits for the SPETG/T40 blend series, the short-range correlation length decreases by 17.6% with increasing ion content, which indicates that the size of the minor phase domain becomes smaller with increasing ion content. In contrast, the long-range correlation length remains relatively constant, while the interfacial surface area of the minor phase (calculated from the volume fractions and values of a_1) increases dramatically with increasing ion content. These trends suggest that the minor phase adopts a more elongated structure and indicate the onset of dual-phase continuity with increasing ion content. A SAXS analysis of the ternary blends containing less than 10% ionomer indicates that these blends behave like the binary blends with varying ion content, as both methods of blending vary only the concentration of ionic functional groups in the blend.

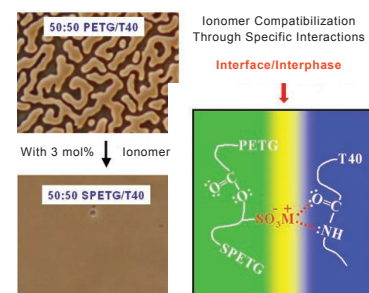


Figure 1. Role of the ionomer (SPETG) in the compatibilization of 50/50 blends of polyamide(T40) and polyester(PETG) binary system. Optical micrographs (left) show that the ionomer yields a profound decrease in dispersed phase size (i.e., below the resolution limit of visible light microscopy/scattering methods).

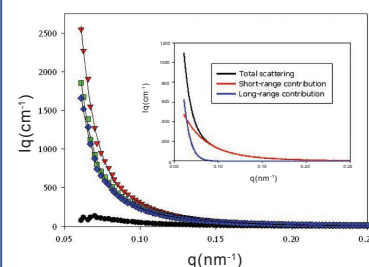


Figure 2. SAXS profiles for the 50/50 polyamide/polyester binary blends: T40/PETG (●), T40/1.9SPETG (▼), T40/3.0SPETG (■) and T40/5.5SPETG (◆). Lines represent the fits to the data from the nonlinear regression analysis using the inset equation. The inset graph shows the relative contribution of the short-range and long-range correlation terms in the nonlinear regression analysis of the measured intensity profiles.